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TOXIC HAZARDS DIVISION

**TOXICOLOGICAL EVALUATION OF  
MATERIALS ASSOCIATED WITH SPACECRAFT**

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## FOREWORD

This paper was presented at the AIAA/ASME Seventh Structures and Materials Conference, Cocoa Beach, Florida, April 1966. The research reported herein was conducted in support of Project 6302, "Toxic Hazards of Propellants and Materials," Task 630201, "Toxicology," in the Toxicology Branch, Toxic Hazards Division, Biomedical Laboratory, Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base, Ohio.

This technical report has been reviewed and is approved.

WAYNE H. McCANDLESS  
Technical Director  
Biomedical Laboratory  
Aerospace Medical Research Laboratories

## ABSTRACT

The Air Force has pursued research in areas of ground support and space cabin toxicology for the past 5 years. Comprehensive treatment of toxicological problems in both areas has revealed the necessity to define human tolerance limits to propellants and other toxic materials for various durations of exposure. Hence, an integrated input of propellant and space cabin material toxicology provides the basis for selection of habitable cabin atmospheres and materials selection criteria. Since such selection procedures are based on both biological and engineering considerations, one cannot disregard the materials selection aspect or evaluate closed system toxicology without consideration of source. The toxic materials in space system atmospheres are determined primarily by the qualitative and quantitative composition of space cabin equipment and the contribution of contaminants by the crew. Materials selection and analytical studies on gas-off products are key considerations in controlling toxic contaminants in a space cabin. Methods are described to determine the composition of cabin materials gas-off products and to biologically test these compounds for their toxicological effects. An attempt is made to correlate the roles of the materials analytical chemist and the toxicologist as a working team to provide meaningful and useful materials selection criteria.

## INTRODUCTION

From the inception of contemplated space flight, the Air Force has been vitally interested in research directed toward providing a safe and habitable environment in the hostile conditions of space. The idea of safely sustaining men in a closed system for long periods of time is not particularly new or unique. The United States Navy has been confronted with this problem for many years and has performed many successful missions requiring prolonged periods of submergence in the nuclear-powered submarine. Approximately 40 years of research effort have been necessary to obtain the underwater capabilities of today's submarine with regard to atmospheric control. Fortunately, the experience and data compiled on submarine habitability problems have direct application to space cabin function. One soon realizes, however, that although the problems are essentially alike in the two systems, environmental control problems in spacecraft will be greatly accentuated by both the external and internal environments of the vehicle. Just as with the submarine, one can anticipate that short manned space missions from 1 to 30 days will present no major difficulties with regard to trace concentrations of toxic contaminants. The only major toxicity hazard for short missions would be of an acute nature only, such as a leaky refrigeration or attitude control system, an accidental spill of some noxious material, or complete failure of the air filtering system. Generally, astronauts could protect themselves from these mishaps by closed-circuit breathing of pure oxygen or, if necessary, by aborting the mission. The cardinal problems, then, would appear to be associated with greater than 2-week missions and also with missions which could not be easily aborted.

For long-term missions, then, one faces the problem of the possibility of intoxication from trace contaminants generated by various space cabin chemicals and materials. In fact, long-term space flights may require closed ecological systems for supplying a habitable environment. This may add chemical, algal, bacterial, and perhaps fungal subsystems to the craft. Since the space cabin will operate between 5- and 14-psi pressure, the problems of boil-off from common substances such as paints, varnishes, adhesives, plastics, oils, solvents, and even metals may become enhanced. Zero-gravity conditions present problems with particulate matter, such as dusts and aerosols, which tends to agglomerate into larger aggregates and to be harmful to both man and filtering systems.

The factors which can aggravate the contaminant concentration are far more numerous than those which can alleviate the problem (table I). The limited volume of usable atmosphere in space systems allows for very little latitude in air pollution control. Air purification and life support equipment are being heavily taxed with increasing mission profile and can, per se, change the total contaminant picture by incomplete processing of toxic materials (ref 1). The state-of-the-art in environmental toxicology does not allow valid predictions of human tolerance to any toxic materials for prolonged continuous exposure (ref 2). Moreover, the bizarre mixture of any contaminants always carries the threat of potentiation of toxic effect by individual constituents within the mixture (ref 3). Exotic environments such as low barometric pressure, single gas oxygen atmosphere, and the multitude of physiological and

psychological stresses are still unknown quantities which can have a profound influence upon man's resistance to chemical insults. Similar to our air pollution problems on earth, freak coincidences of relatively harmless factors could lead to severe biological embarrassment. It is also quite obvious that the problems on nuclear submarines are far less serious than in spacecraft environment. Finally, truly uninterrupted 90-day continuous exposure to contaminant concentrations not exceeding the Threshold Limit Value has resulted in 100% mortality of animals with certain chemicals (ref 4).

TABLE I

IMPORTANT FACTORS INFLUENCING  
ATMOSPHERIC CONTAMINATION

Aggravating	Beneficial
Continuous Generation and Exposure	Leak Rate of Cabin Materials Selection
*Reduced Pressure	Preconditioning of Materials
*Volume/Man Ratio	
*Power and Weight Limitation	
Filter Characteristics	
Complexity of Contaminants	
*Multi-Stress Environment	
*Escape Lead Time	
*Not significant in nuclear submarines.	

Until 1964, the question of whether a man could safely live in an environment of 100% oxygen at 5 psi for longer than 30 days was unanswered. It had been known for quite some time that pure oxygen by itself was toxic when breathed for 1 to 2 days under sea level pressures. It was also surmised that oxygen toxicity might cause certain changes in the presence of other trace contaminants which were also irritants. This, in effect, would enhance the toxicity of these contaminants. Data which proved or disproved these synergistic effects have been unavailable until just recently. When toxicologists were asked to provide information regarding the toxic effects of contaminants in an oxygen-rich environment for long continuous periods of time, it was soon evident that there were no data available, and also no equipment at hand with which to gather such data. Consequently, scientists of the Toxic Hazards Division, Aerospace Medical Research Laboratories, started work in this unique field of trace contaminant toxicology.

## RESEARCH FACILITY

### TOXIC HAZARDS RESEARCH UNIT

A Toxic Hazards Research Unit (ref 5) has been designed, constructed, and is currently operating to study the toxic hazards of trace contaminants at reduced atmospheric pressure. This unit was designed by Dr. Anthony A. Thomas, Chief of the Toxic Hazards Division, and was constructed by Aerojet-General Corporation, Azusa, California, under a \$1.5 million, 3-year contract with the Air Force Systems Command. Both Aerospace Medical Research Laboratories and Aerojet personnel staff the research facility.

This unique inhalation exposure unit has the capacity to perform toxicological research on a large number of animals at simulated atmospheric compositions of typical space cabin conditions, that is, at reduced atmospheric pressures (5-14 psi) and in either single (oxygen) or mixed gas (oxygen/nitrogen) atmospheres. The primary mission of this new facility is to study the effects of truly uninterrupted, prolonged continuous exposure to various trace contaminants under a single gas oxygen atmosphere. The unit is capable of performing experiments continuously from 2 weeks to more than 1 year.

The research unit consists of four inhalation exposure chambers (Thomas Domes). Each dome is 12 feet in diameter and 9 feet in height. Glass paneling permits unrestricted visual and photographic observation of animal or human activity in any direction. This greatly facilitates the use of trained animals for psychopharmacological evaluation. Behavioral and biological instrumentation can be placed within the domes. Forty pairs of shielded cable are available for transmission of monitoring signals. Each dome may be elevated from its base to aid in loading and unloading of equipment and animals and also to provide a quick escape mechanism in case of fire or other mishap. Daily servicing is accomplished through a vertical airlock system. This allows for undisturbed exposure conditions.

All vital systems including vacuum pumps, air compressors, air conditioning, and electrical power generators are present in duplicate to act as back-up in case of failure of any individual unit. The system is a dynamic flow configuration in which oxygen and any contaminant are introduced to a dome through a single diffuser pipe, and the flow proceeds to an O-ring located in the floor of the dome, is filtered, and is exhausted to the outdoors. The dynamic flow system prevents pile-up of contaminants and volatile animal excrete and is maintained at a constant five complete changes per hour.

All entries into the domes by humans are monitored by an intercommunication system which is backed up by walkie-talkie and a loudspeaker system. Three persons are necessary to operate each dome during entry for emergency purposes. In case of accidents, a person either inside or outside of the dome can "dive" the test chamber to atmospheric level, and the dome can be immediately lifted (10 seconds) for rapid escape. An automatic and manual water deluge system is available in case of fire.



## Psychopharmacology

One of the domes has been equipped with 12 behavioral units for use in testing performance changes of monkeys exposed to selected contaminants. Each of the 12 units consists of cages equipped with a performance panel and the necessary solid-state programming equipment, located outside of the dome, to present the animal with the proper tasks. The programmer and accessories are attached to each unit within the dome via cables and connectors which can function at altitude without leaking or presenting a fire hazard.

## Materials Testing and Generating System

One of the domes has been equipped to test the inhalation toxicity of space cabin materials using rodents as the test animals. The dome is used as an environmental envelope containing a completely closed system in which the rodents are continuously exposed to recirculating trace contaminants. The contaminants are gassed-off in ovens maintained at 180 F and in 100% oxygen or in a mixed gas atmosphere, as required. The materials flow into a cage system containing the rodents and back through the ovens after being scrubbed for carbon dioxide and excess water vapor. Materials to be tested are placed in the ovens roughly in the amounts in which they will be used within a space cabin and under conditions which might be expected to give maximum surface area. Early screening is performed using groups of materials. If toxicological signs are observed, as evidenced by decrease in body weight of the animals or other gross signs of toxicity, further tests are done to determine which material or materials in the group are responsible.

## Analytical

To test the absolute composition of the atmospheres in the Thomas Domes, a battery of highly specialized analytical instrumentation is available. This includes a Time-of-Flight mass spectrometer and necessary accessories to sample gases, liquids, and particulates simultaneously. In addition, gas chromatographs with flame ionization, thermal conductivity and electron capture detectors as well as infrared spectrometers, spectrophotometers (visible and ultraviolet), and spectrofluorophotometers are available as necessary to evaluate environmental species. These instruments are also used to provide fingerprinting of gas-off products from single materials exposed to 100% oxygen in a container evacuated to 5 psia, or to air under ambient conditions.

## RESEARCH OBJECTIVES

The U.S. Air Force mission is to provide both fundamental experimental data on space cabin toxicology and a quick reaction capability in the toxicological qualification of space cabin materials. A cost sharing U.S. Air Force-National Aeronautics and Space Administration research effort is currently exploring the following fundamental and practical questions:

1. Does 5 psi, single gas, oxygen atmosphere cause pulmonary irritation or functional impairment during long-term 14-day to 1-year exposure?
2. Will a 5 psi, single gas, oxygen atmosphere influence tolerance to toxic materials, especially to pulmonary irritants and systemic poisons?
3. Will a 5 psi, mixed gas, 70% oxygen—30% nitrogen atmosphere influence tolerance to toxic materials?
4. Should space cabin materials be screened for toxic properties singly or in mixtures?
5. Are there differences in gas-off properties of space cabin materials at 5 psi, in single versus mixed gas atmospheres?
6. What are the chemical gas-off profiles of compounds exposed to single and mixed gas atmospheres for 30, 60, and 90 days?

## RESULTS AND DISCUSSION

### OXYGEN TOXICITY

The facility has been in constant use since September 1964. During this time, one of the domes has been devoted to the problem of studying the effects of 100% oxygen at 5 psia on monkeys, dogs, rats, and mice. The purpose of the tests was to explore the suitability of this atmosphere for long mission durations and to differentiate the effects of contaminants from the effects of contaminants and oxygen combined. The longest continuous exposure in space cabin toxicology research was completed on 4 December 1965. The animal complement consisted of 40 mice, 65 rats, 8 beagle dogs, and 4 rhesus monkeys and the exposure lasted 236 days. Examination of the animals by pathological and clinical observation showed that this environment was, for the most part, innocuous. Other experiments that were run for 16 and 90 days, from which tissues were taken for electron microscopy studies, indicated that there were some changes at the cellular level which tended to revert to normal with time.

Experiments using 100% oxygen at 760, 720, 700, 650, and 600 mm of mercury were performed for comparative purposes. It was found that toxicity decreased as total  $pO_2$  decreased. In addition, the manner in which toxicity became evident changed as the altitude increased. Animals exposed at 760 mm Hg pressure succumbed to the environment within a 3-day period and died from pulmonary edema and hemorrhage; those exposed to 700 or less mm Hg pressure showed marked decrease in mortality and evidence of pulmonary fibrotic change rather than primary hemorrhagic changes.

Psychopharmacological testing, using 12 trained monkeys at 5 psia, 100% oxygen for 90 days, showed no decrement of performance to either continuous or discrete avoidance tasks.

The foregoing studies clearly indicate the feasibility of applying 5 psi oxygen atmospheres for at least 90-day mission durations in orbital systems.

#### EFFECTS OF 5 PSI, 100% OXYGEN UPON TOXICITY

These studies were performed for 14-day periods using either ozone, nitrogen dioxide, or carbon tetrachloride as the toxic contaminant (ref 6). The results are shown in tables II and III.

TABLE II  
CONTAMINANT TOXICITY AT 5 PSI, 100% OXYGEN

Continuous Exposure			Cumulative Mortality			
Duration	Concentration	Compound	Mice	Rats	Dogs	Monkeys
90 day	100%	Oxygen	3/40	9/40	0/4	0/4
14 day	630.00 mg/m <sup>3</sup>	CCl <sub>4</sub> + O <sub>2</sub>	39/40	0/50	0/8	0/4
	80.00 mg/m <sup>3</sup>	" " "	0/40	7/50	0/8	0/4
	100%	Control O <sub>2</sub>	0/40	8/40	0/2	0/2
14 day	84.6 mg/m <sup>3</sup>	NO <sub>2</sub> + O <sub>2</sub>	40/40	37/50	7/8	4/4
	38.00 mg/m <sup>3</sup>	" " "	5/40	0/50	0/8	2/4
	17.00 mg/m <sup>3</sup>	" " "	0/40	0/50	0/8	0/4
14 day	100%	Control O <sub>2</sub>	0/40	0/40	0/4	0/4
	14.7 mg/m <sup>3</sup>	O <sub>3</sub> + O <sub>2</sub>	...	...	6/8	0/4
	7.8 mg/m <sup>3</sup>	" " "	33/40	45/50	2/8	0/4
	3.92 mg/m <sup>3</sup>	" " "	0/40	0/50	1/8	0/4
	1.96 mg/m <sup>3</sup>	" " "	0/40	0/50	0/8	0/4
	100%	Control O <sub>2</sub>	3/20	2/20	0/4	0/4

Comparative results indicate a number of interesting and important bits of data. It is quite evident that there are species differences to various concentrations of these compounds. That is, the dog is more susceptible to the pulmonary irritant effects of ozone than is the monkey and the reverse appears true for NO<sub>2</sub>. All animals exposed to 2 mg/m<sup>3</sup> ozone survived under 5 psi 100% oxygen conditions. The Threshold Limit Value (TLV) for an 8-hour day for man is 0.2 mg/m<sup>3</sup> ozone.

The TLV for  $\text{NO}_2$  is  $9 \text{ mg/m}^3$  and  $65 \text{ mg/m}^3$  for  $\text{CCl}_4$ . The pulmonary irritants are less toxic at 5 psi, 100% oxygen than in ambient air. Further, 70%  $\text{O}_2$ —30%  $\text{N}_2$  at 5 psi appears to be less effective than 100% oxygen. In contrast, the systemic poison,  $\text{CCl}_4$ , did show slightly more toxicity at altitude than on the ground. This may be explained by early reversible liver changes seen during 2-week exposure to oxygen (ref 7) and increased susceptibility of liver to  $\text{CCl}_4$  under these conditions.

TABLE III

COMPARATIVE TOXICITY DURING 14-DAY EXPOSURE

Exposure	5-psi, 100% $\text{O}_2$	5-psi, 70% $\text{O}_2$ —30% $\text{N}_2$	Ambient Air
<hr/>			
8 mb/ $\text{m}^3$ , $\text{O}_3$			
<hr/>			
Dogs	2/8	6/8	5/5
Monkeys	0/4	1/4	2/4
Rats	45/50	45/50	50/50
Mice	33/40	33/40	34/40
38 mg/ $\text{m}^3$ , $\text{NO}_2$			
<hr/>			
Dogs	0/8	0/8	0/5
Monkeys	2/4	3/4	4/4
Rats	3/50	1/50	5/50
Mice	0/40	8/40	2/40
<hr/>			

#### SPACE CABIN MATERIAL TOXICITY SCREENING

Groups of 10-14 space cabin materials have been screened as outlined previously. To date, about 80 of some 400 materials have been evaluated for 1-week exposures. One of four groups produced a change in weight gain of the rats used in the test. Our early data indicate that 1 week is not long enough to extrapolate trace contaminant toxicity to the long mission profile. It has proved to be much better than odor or other such criteria for acceptability testing, but most of the testing henceforth will entail mixtures tested for 60 days.

#### SPACE CABIN MATERIAL OFF-GASSING

Under U.S. Air Force Contract AF 33(615)-1779 with Monsanto Research Corporation, 55 materials were stored in glass chambers for 30, 60, and 90 days at 23-25 C, 20-40% relative humidity, in air at 1 atmosphere, and at 5 psia in 100%  $\text{O}_2$ . General findings were as follows. There were considerable amounts of gas-off from some materials prepared immediately before testing. They included coatings, paints, and adhesives. There was little, if any, gas-off from fabricated materials such as

polycarbonates, polyvinylfluorides and nylon-based materials. The major gas-off products came from solvents, plasticizers and monomers. The gas-off products were different, in many cases, at 1 atmosphere from those at 5 psia, 100% oxygen. That is, the chemical profiles changed under 100% O<sub>2</sub> conditions. Some coatings desorbed considerable quantities of carbon monoxide. Others gave off trimethyl silanol and low molecular weight methyl siloxane polymers. Quantitative analysis was greatly influenced by nonuniformity of sample lots, changes in sample homogeneity, freshness of sample, free surface area, adsorptive characteristics of the encapsulating chamber and sampling methods. The data gathered under this and other efforts of this type will ultimately provide the toxicologist with lists and chemical "fingerprints" of space cabin atmosphere profiles. We shall then be better able to evaluate the toxic parameters of individual components as necessary.

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